

UK Patent Application (19) GB (11) 2 020 225 A

(21) Application No 7911546
(22) Date of filing 3 Apr 1979
(23) Claims filed 3 Apr 1979
(23) Claims filed 6 Jul 1979
(30) Priority data
(31) 15202/78
(32) 18 Apr 1978
(33) United Kingdom (GB)
(43) Application published
14 Nov 1979
(51) INT CL²
B29D 23/04
(52) Domestic classification
B5B 35Y 360 FC
(56) Documents cited
None
(58) Field of search
B5B
(71) Applicant
Du Pont of Canada
Limited, P.O. Box 660,
Montreal, Quebec,
Canada, H3C 2V1
(72) Inventor
David Alan Harbourne
(74) Agent
Mewburn, Ellis & Co.

(54) Manufacture of Film From Partially Crosslinked Polyethylene

(57) A blown film process is disclosed, in which polyethylene film with a reduced transverse direction tear strength is produced. The process has a blow-up ratio in the range of 1.2:1 to 4:1 and a MD draw down ratio in the range of 2 to 60. The polyethylene is partially crosslinked and extruded into a film between 10 μm and 100 μm thick. The polyethylene before crosslinking has a density of between

0.940 g/cm³ and 0.970 g/cm³, and a melt index of between 0.2 g/10 min and 15.0 g/10 min. The crosslinking should be sufficient to cause the film to have a substantial reduction in the Elmendorf tear strength in the transverse direction as against a film made under the same conditions using the same polyethylene not crosslinked. Any known crosslinking method may be used, examples of methods and sufficient doses are disclosed, as are preferred ranges for other variables.

ERRATUM

SPECIFICATION NO 2020225A

Front page, Heading (71) Applicant *delete* Du Pont of Canada Limited *insert* Du Pont Canada Inc.

THE PATENT OFFICE
4 January 1980

Bas 73202/19

GB2 020 225 A

SEE ERRATA SLIP ATTACHED

(12) UK Patent Application (19) GB (11) 2 020 225 A

(21) Application No 7911546
(22) Date of filing 3 Apr 1979
(23) Claims filed 3 Apr 1979
(23) Claims filed 6 Jul 1979
(30) Priority data
(31) 15202/78
(32) 18 Apr 1978
(33) United Kingdom (GB)
(43) Application published
14 Nov 1979
(51) INT CL²
B29D 23/04
(52) Domestic classification
B5B 35Y 360 FC
(56) Documents cited
None
(58) Field of search
B5B
(71) Applicant
Du Pont of Canada
Limited, P.O. Box 660,
Montreal, Quebec,
Canada, H3C 2V1
(72) Inventor
David Alan Harbourne
(74) Agent
Mewburn, Ellis & Co.

(54) **Manufacture of Film From
Partially Crosslinked Polyethylene**

(57) A blown film process is disclosed, in which polyethylene film with a reduced transverse direction tear strength is produced. The process has a blow-up ratio in the range of 1.2:1 to 4:1 and a MD draw down ratio in the range of 2 to 60. The polyethylene is partially crosslinked and extruded into a film between 10 μm and 100 μm thick. The polyethylene before crosslinking has a density of between

0.940 g/cm³ and 0.970 g/cm³, and a melt index of between 0.2 g/10 min and 15.0 g/10 min. The crosslinking should be sufficient to cause the film to have a substantial reduction in the Elmendorf tear strength in the transverse direction as against a film made under the same conditions using the same polyethylene not crosslinked. Any known crosslinking method may be used, examples of methods and sufficient doses are disclosed, as are preferred ranges for other variables.

123456789

SEE ERRATA SLIP ATTACHED

SPECIFICATION

Manufacture of Film from Partially Crosslinked Polyethylene

The present invention relates to an improved process for the manufacture of polyethylene film. In particular the present invention relates to a process for the manufacture of film from polyethylene that 5 has been partially cross-linked.

Methods for the manufacture of polyethylene film are known. One of these methods is the so-called conventional blown film process, an embodiment of which is described in Canadian Patent 460 963. In a conventional blown film process molten polyethylene is extruded through an annular die having a die gap between about 0.38 mm and 1.30 mm. The tubular molten polyethylene so formed is 10 continuously withdrawn from the die, cooled to the solid state, flattened and wound up on a roll. The molten polyethylene is usually cooled, by means of an air ring, soon after the polyethylene is extruded through the die. The tubular film may be expanded to the desired extent by means of, for example, a controlled pressure of gas inside the tubular film.

The term "blow-up ratio" as used herein means the ratio of the diameter of the solidified 15 expanded film tubing to the diameter of the annular die, in a blown film process.

The term "MD draw down ratio" as used herein means the ratio of the width of the die gap of the annular die to the product of the blow-up ratio and the thickness of the film. The thickness of the film is measured subsequent to blowing the film.

The term "frost line" as used herein refers to a narrow zone on the extruded polyethylene film 20 within which the polyethylene changes from a molten or semi-molten state to a solid state. With polyethylenes which do not contain opacifying fillers, e.g. pigments, the change of state causes a change in optical properties of the film, which is readily observable by the naked eye. The term "frost line height" refers to the distance between the annular die and the frost line.

One of the important factors in determining the economics of the manufacture of polyethylene 25 film is the rate at which the film is produced. In blown film processes a high rate of production is most usually accomplished by producing the film using a large diameter die and a low blow-up ratio, e.g., in the range of about 1.5 to 2.0. In addition wide die gaps may sometimes be used to increase the rate at which the film is produced.

Such blown film processes, when used to manufacture high density polyethylene film i.e. film 30 made from polyethylene having a density in the range of 0.940 to 0.970 g/cm³, tend to result in film having a relatively low tear strength in the machine direction (MD) and relatively high tear strength in the transverse direction (TD). The term "tear strength" as used herein is the Elmendorf tear strength as measured by the procedure of ASTM D-1922.

Films having high TD tear strengths are undesirable for use in applications where tearing or 35 cutting in the transverse direction is important, for example, in the manufacture of bags e.g. self-opening square or notion and millinery bags, or for the ease of opening sealed packages e.g. cereal or chip liners. In such applications and in those where it is desired to replace paper by polyethylene film it is often desirable that the film tear as easily in the transverse direction as it does in the machine direction i.e. provide a film that has a "balanced tear", and that the tear strength be low. It is known 40 that the TD tear strength of polyethylene film may be lowered by increasing the frost line height, increasing the blow-up ratio, decreasing the die gap, and incorporating inorganic fillers into the polyethylene, among other techniques. However some of these techniques, for many polyethylene films, tend to lower the rate of production of film and/or sacrifice quality related properties of the film e.g. film gauge uniformity.

An increase in melt index of the polyethylene, as measured by the procedure of ASTM D-1238 45 (condition E) also permits higher extrusion rates than are possible for polyethylene of lower melt index, and aids in reducing the TD tear strength of film produced therefrom. However, as the melt index is increased, the melt strength of the polyethylene tends to be decreased and may make the blown film process unstable and/or the impact strength of the film produced therefrom unacceptably low for many 50 applications.

A method for improving melt strength and melt extensibility of polyethylene having a density of 0.95—0.97 g/cm³ and a melt index of 3—13 g/10 min is described in U.S. Patent 3,563,870. The method involves exposing the polyethylene to 0.05 to 0.15 megarad (5000 to 15000 J/kg) of high energy radiation.

An improved process for the manufacture of polyethylene film, especially with regard to the tear of 55 such film in the transverse direction has now been found.

Accordingly, the present invention provides a blown film process in which partially cross-linked polyethylene is extruded into film having a thickness in the range of 10 to 100 μ m, said process having a blow-up ratio in the range of 1.2:1 to 4:1 and a MD draw down ratio in the range of 2 to 60, said 60 polyethylene having had before cross-linking, a density in the range of 0.940 to 0.970 g/cm³ and a melt index in the range of 0.2 to 15.0 g/10 min and having been cross-linked to an extent sufficient to provide, after extrusion in said blown film process, a film having an Elmendorf tear strength in the transverse direction substantially less than the Elmendorf tear strength in the transverse direction of a

film produced from the polyethylene, before cross-linking, under the same conditions as the film produced from the partially cross-linked polyethylene.

It is preferred that the film thickness be in the range of 15 to 100 μm and the MD draw down ratio be in the range of 3 to 60.

5 In a preferred embodiment of the process of the present invention the polyethylene is cross-linked to an extent sufficient to provide a film having an Elmendorf tear strength in the transverse direction in the range of 0.4 to 10 g/ μm , especially in the range of 0.4 to 2.5 g/ μm , the film being extruded to a thickness between 25 and 35 μm . 5

10 In another embodiment the polyethylene is cross-linked to an extent sufficient to provide a film having an Elmendorf tear strength in the transverse direction in the range of 10 to 150 g, the film being extruded to a thickness between 35 and 100 μm . 10

15 The MD drawn down ratio is most preferably in the range of 6 to 40. The preferred range for the blow up ratio is from 1.2:1 to 2.5:1. Frost line height is preferably in the range of 10 to 66 cm, most preferably from 10 to 40 cm.

20 15 In yet another embodiment the polyethylene is a homopolymer of ethylene, especially a linear homopolymer of ethylene, or a copolymer of ethylene and a higher α -olefin or mixture thereof.

25 In the process of the present invention molten polyethylene is extruded through an annular die. The extruder and die must be capable of supplying a substantially homogeneous stream of molten polymer to the lips of the die. In order to control the process and to facilitate the manufacture of a 20 uniform product, the variation in thickness of the molten polymer extruded through the lips of the die preferably is low. The temperature of the molten polymer when it is extruded through the annular die, while being important in maintaining the frost line at a desired position, may depend in part on the melt flow and melt strength properties of the polymer. The term "melt flow properties" of the polymer is a general term used to describe the processability of a polymer at different temperatures. For example, 25 because of differences in molecular weight, molecular weight distribution, degree of branching and the like, it may be necessary to process polyethylenes of different types at different temperatures in order to achieve, for each particular polyethylene, a uniform flow of polymer from the extruder. The term "melt strength" relates to the strength of the polymer in a molten state especially with regard to the ability of the polymer to support its own weight without significant distortion of, for example, the 30 tubular film of molten polymer. 30

35 Conventional blown film processes for the manufacture of polyethylene film are normally operated with the film being extruded in a vertical direction. After extrusion of the molten polymer through an annular die, the tubular film is expanded to the desired extent, cooled or allowed to cool and flattened. The tubular film is flattened by passing the film through a set of nip rolls. A positive pressure 35 of gas, for example air or nitrogen, is maintained inside the tubular film so that the desired degree of expansion of the tubular film occurs.

40 The polymer used in the process of the present invention is polyethylene, having a density in the range 0.940—0.970 g/cm³. Such polyethylene may be a homopolymer of ethylene or a copolymer of ethylene and a higher α -olefin having 4—10 carbon atoms e.g. butene-1, hexene-1 or octene-1. The 40 polyethylene may be manufactured by processes known to those skilled in the art. In a preferred embodiment the homopolymer is a linear homopolymer of ethylene. In a particular embodiment the polyethylene is a homopolymer of ethylene or a copolymer of ethylene and a higher α -olefin obtained from a low pressure polymerization process in which the monomers are polymerized in the presence of a co-ordination catalyst. In a preferred embodiment the polymerization process is a solution 45 polymerization process. 45

50 In order to obtain the improvement of the present invention, the polyethylene is partially cross-linked. The amount of cross-linking is such that the polyethylene so obtained is partially cross-linked i.e. more cross-linked than the untreated polymer, but not cross-linked to the extent that the polymer is no longer thermoplastic. 50

55 Cross-linking of the polyethylene is carried out to an extent sufficient to effect a substantial reduction in the TD tear strength of the film made by the blown film process of the present invention. There appears to be a threshold amount of cross-linking below which the TD tear strength of the film produced is substantially unaffected. The amount of cross-linking required depends in a large part upon the level of TD tear strength and the imbalance of MD and TD tear strengths of film made from the uncross-linked polyethylene. 55

60 For example an unmodified polyethylene extruded at a high MD draw down ratio and at a low blow-up ratio would require more cross-linking than the same unmodified polyethylene extruded at a lower MD draw down ratio and a higher blow-up ratio in order to effect a given low level of TD tear strength or a given extent of balance of MD to TD tear strengths. Similarly a polyethylene containing a 60 filler e.g. ground mica would require less cross-linking than an unfilled polyethylene would to effect the same low level of TD film tear strength. It may be seen from the examples hereinafter that substantial changes in Elmendorf TD tear strength may be effected by cross-linking the polyethylene beyond the threshold value required for each polyethylene. For example, it may be seen that substantial reductions, to 50% and less of the value of the Elmendorf TD tear strength of the blown film made from the uncross-linked polyethylene, may be effected by partial cross-linking. 65

A number of methods are known for the cross-linking of polymers, including polyethylene. Such methods may in general be controlled so as to effect the partial cross-linking of the polyethylene used in the process of the present invention. Examples of methods of cross-linking of polyethylene are the use of organic peroxides e.g. di- α -cumyl peroxide, tertiary butyl peroxide, α, α' -bis(t-butylperoxy) diisopropyl benzene, and 2,5-dimethyl 2,5-di(t-butylperoxy) hexyne-3, the use of other organic cross-linking agents e.g. α, α' -azo-diisobutyronitrile, the use of silane cross-linking agents e.g. as disclosed in U.S. Patents 3,075,948 and 3,646,155, and the use of high energy radiation. Representative of such radiation are beta rays, gamma rays, X-rays, electron beams, neutrons and the like. 5

A variety of techniques are known in the art for effecting cross-linking of polyethylene. If the 10 techniques involve incorporation of a cross-linking agent e.g. organic peroxide, other organic cross-linking agents, or silane cross-linking agents, in the polyethylene, the agent may be metered into the extruder being used for the manufacture of the polyethylene film, thereby directly forming the films of partially cross-linked polyethylene. Alternatively, the agent may be metered into the polyethylene in an 15 extruder and with or without cross-linking the polyethylene, forming pellets of the polyethylene which are subsequently extruded into film. Instead of metering the agent into the polyethylene in an extruder, the agent may be coated onto and/or impregnated into pellets of the polyethylene. 15

If irradiation techniques are used, the shape or form of the polyethylene during irradiation is not particularly critical. However, it is generally desirable to carry out irradiation on polyethylene in the form of particles having diameters of less than six millimetres e.g. powders, beads, pellets or the like. 20

During irradiation, steps should be taken to ensure that the polyethylene is treated in a reasonably uniform manner and especially so that gel is not formed in the film subsequently manufactured from the treated polyethylene. 20

Other additives, for example, fillers, especially lamellar fillers e.g. mica and talc, pigments, stabilizers and antioxidants may be incorporated into the polyethylene. 25

Pigments may, depending on the pigment type, form, size and concentration in the polyethylene, modify the properties of the films obtained by the process of the present invention. 25

However some additives, especially antioxidants may have an effect on the partial cross-linking of the polyethylene and on the long term stability of films produced therefrom. 30

The uniformity of the partial cross-linking of the polyethylene should be such that no significant amount of gel is formed in the polymer and hence in the film subsequently manufactured. 30

It has also been found that the processability in a blown film process of certain uncross-linked polyethylene resins may be improved and the TD tear strength of the film resulting therefrom lowered by blending the uncross-linked polyethylene with a partially cross-linked polyethylene according to the present invention. As will be readily understood the higher the TD tear strength of blown film resulting 35

from extrusion of the uncross-linked polyethylene the greater the concentration of partially cross-linked polyethylene in the blend would be required. It has been found that no significant benefit is obtained, even with uncross-linked polyethylenes which produce films of lower TD tear strength, unless the concentration of partially cross-linked polyethylene in the blend is at least 20% by weight i.e. there is at most 80% by weight of uncross-linked polyethylene in the blend. 35

Blending of the two polyethylenes provides further versatility to the present invention. It may be used for example for making a film having an inorganic filler in which a "masterbatch" of polyethylene compounded with the inorganic filler is blended with a second polyethylene prior to extrusion into film. The second polyethylene may, advantageously, be a partially cross-linked polyethylene as used in the present invention. 40

As stated hereinabove, the process of the present invention results in the formation of polyethylene film of improved properties especially with respect to the Elmendorf tear strength in the transverse direction. Relatively low values of such tear strength e.g. values of less than 2.5 g/ μ m are beneficial especially in the cutting of such film during packaging operations. Films of 25 to 35 μ m thickness and an Elmendorf tear strength in the transverse direction less than about 2.5 g/ μ m are 45

easily torn by hand in the transverse direction and demonstrate good cutting performance on certain packaging machines which normally are fed with paper as the packaging material. For use in such packaging machines, it may be advantageous, in addition to selecting a film having a low Elmendorf TD tear strength, to select a film having a low MD elongation, i.e. less than about 200% and more particularly less than 100%. 50

MD elongation values as shown herein were measured by the procedure of ASTM 1248-60T/D 412-51T, using a D412-68 Type C sample configuration at an extension speed of 50.8 cm/min. 55

The present invention is illustrated by the examples following.

Dart drop was measured by the procedure of ASTM-D 1709, with the exception that the dart was 38.1 mm in diameter and had a nylon head, and the dart was dropped from a height of 25.4 cm. 60

Example 1

Samples of polyethylene were irradiated with gamma rays or electron beams to partially cross-link the polyethylene. The polyethylenes so treated were extruded into film using conventional film blowing apparatus. TiO_2 pigment in the form of Ampacet[®] 11200 concentrate was added at the extruder to provide a TiO_2 concentration in the film of 2% wt/wt. The annular die used had a diameter 60

10.160 cm and the die gap was 1.016 mm. The operating conditions of the blown film process were adjusted to form a film about 25.4 μm in thickness.

5 Polyethylene type A used in Runs 1 to 3 was an ethylenebutene-1 copolymer having, prior to irradiation, a broad molecular weight distribution, a density of 0.950 g/cm³ and a melt index of 0.45, available from Du Pont of Canada Ltd. Montreal, Canada, under the Trade Mark Sclair® and designated type 58A. 5

10 Polyethylene Type B, used in Runs 4 to 12 was an ethylenebutene-1 copolymer having, prior to irradiation, an intermediate molecular weight distribution, a density of 0.945 g/cm³ and a melt index of 0.26 g/10 min available from Du Pont of Canada Ltd. under the Trade Mark Sclair and designated Type 10 16A. 10

Polyethylene type C, used in Runs 13 and 14 was an ethylene homopolymer having, prior to irradiation, a narrow molecular weight distribution, a density of 0.960 g/cm³ and a melt index of 7.3 g/10 min.

15 Polyethylene type D, used in Run 15 was an ethylene homopolymer having an intermediate molecular weight distribution, a density of 0.960 g/cm³ and a melt index of 1.67 g/10 min. 15

Further experimental details and the results obtained are shown in Tables I and II.

Table I

Run	PE ¹	Irr. ²	Dosage ³ (J/g)	Melt Index* (g/10 min)	Frost Line Height (cm)	Blow-up Ratio	20
1**	A	none	0	0.45	30	3:1	
2	A	γ	27.0	0.15	23	3:1	
3	A	E	63.0	0.04	23	3:1	
4**	B	none	0	0.26	23	3:1	25
5	B	E	25.0	0.10	23	3:1	
6	B	E	38.0	0.06	23	3:1	
7**	B	None	0	0.26	66	3:1	
8	B	E	25.0	0.10	66	3:1	
9	B	E	38.0	0.06	66	3:1	30
10**	B	none	0	0.26	66	2:1	
11	B	E	25.0	0.10	66	2:1	
12	B	E	38.0	0.06	66	2:1	
13	C	γ	125	1.35	66	2:1	
14	C	γ	125	1.35	66	3:1	35
15**	D	none	0	1.67	30	3:1	

1 PE=polyethylene

2 Irr=Irradiation

3 γ =gamma ray

40 4 E=electron beam

* Melt index of partially cross-linked polyethylene determined after cross-linking but prior to extrusion into film

** Comparison runs. The polyethylene in these runs had not been cross-linked.

Table II

5	Elmendorf Tear Strength (g/μm)		Dart Drop F_{50} (g/μm)	MD Elongation %	5
	Run	Machine Direction			
	1*	0.76	45	20	530
	2	0.84	35	1.3	640
	3	0.64	1.2	≤1.3	49
	4*	0.52	49	2.0	405
10	5	0.52	9.7	5.0	530
	6	0.60	6.8	3.3	510
	7*	0.68	22	14.0	460
	8	0.88	2.5	8.0	720
	9	0.84	1.2	5.7	730
15	10*	0.52	36	12.0	—
	11	0.72	3.6	7.8	630
	12	0.64	3.1	6.0	570
	13	0.69	1.7	2.4	820
	14	0.86	1.3	2.3	690
20	15*	0.72	6.9	2.9	760

* Comparison runs. The polyethylene in these runs had not been cross-linked.

The results show that partial cross-linking of the polyethylene, as indicated by melt index resulted in the manufacture of polyethylene film having reduced Elmendorf tear strength in the transverse direction. It may also be seen from Runs 1, 2 and 3 that there appears to be a threshold level of 25 irradiation, over which a polyethylene must be irradiated in order to significantly alter the properties, particularly of Elmendorf tear strength in the transverse direction, of the film produced therefrom.

A comparison of Run 15 with the other Runs appears to indicate that a high melt index *per se* is insufficient to provide a film having a very low Elmendorf TD tear strength.

Example II

30 The procedure of Example I was repeated except that the polyethylenes were partially cross-linked using an organic peroxide viz. 2,5-dimethyl 2,5-di(t-butylperoxy)hexyne-3, as the cross-linking agent. Runs 18 to 20 did not have the TiO_2 pigment added. 30

Cross-linking of the polyethylenes was accomplished in a two-stage procedure. The polyethylene to be partially cross-linked i.e. base resin was compounded at a temperature of less than about 150°C. 35 with the requisite amount of concentrate of 2,5-dimethyl 2,5-di(t-butylperoxy)hexyne-3 in low density polyethylene. The amount of low density polyethylene introduced was not sufficient to cause any noticeable difference in the behaviour of the base resin. A second, high temperature e.g. about 200°C, compounding was then employed to completely decompose the peroxide and bring about the required extent of cross-linking of the base resin, after which the partially cross-linked polyethylene was 40 extruded into pellets suitable for further processing into film. The masterbatch of 2,5-dimethyl 2,5-di(t-butylperoxy)hexyne-3 in low density polyethylene used to prepare the partially cross-linked polyethylenes used in Runs 17, 19, 20, 22 and 23 had 0.9% wt. of the peroxide/wt of polyethylene.

Polyethylene type B used in Runs 16 and 17 was Sclair 16A polyethylene resin as in Example I.

Polyethylene type E used in Runs 18 and 19 was blend of 3 parts by weight of an ethylene 45 homopolymer having prior to cross-linking a broad molecular weight distribution, a density of 0.960 g/cm^3 and a melt flow index of 0.75 g/10 min, available from Du Pont of Canada Ltd. under the Trade Mark Sclair and designated type 19A, to 1 part by weight of a second polyethylene having a melt index of 4.7 g/10 min. an intermediate molecular weight distribution and a density of 0.952 g/cm^3 containing 40% by weight of ground mica. Only the Sclair 19A polyethylene of the polyethylene type E blend was 50 partially cross-linked using the peroxide. The melt index shown for Run 19 indicates the melt index determined for the cross-linked Sclair 19A polyethylene and not for the polyethylene type E blend.

Polyethylene type F used in Run 20 was a blend of 3 parts by weight of an ethylene homopolymer having prior to cross-linking a narrow molecular weight distribution, a density of 0.96 g/cm^3 and a melt index of 7.3 g/10 min to 1 part by weight of a second polyethylene, having a melt index of 4.7 g/10 min, an intermediate molecular weight distribution and a density of 0.952 g/cm^3 , containing 40% by weight of ground mica. Only the homopolymer of the polyethylene type F blend was partially cross-linked using the peroxide. The melt index shown for Run 20 indicates the melt index of the cross-linked homopolymer and not for the polyethylene type F blend. 55

The operating conditions of the blown film process of Runs 16, 17 and 21 were adjusted to form 60 films of about 25.4 μm in thickness and of Runs 18 to 20 to form films of about 43.2 μm in thickness. Because of the surface roughness of the films produced from polyethylene types E and F the film thicknesses were calculated after determining the density and the weight of a known area of film.

Further experimental details and the results obtained are shown in Tables III and IV.

Table III

PE ¹ Peroxide		Concentration ppm by weight	Melt Index* (g/10 min)	Frost-line Height (cm)	Blow-up Ratio	
Run	Type	None	0.28	30	3:1	5
5	16**	B	210	0.05	30	3:1
	17	B	None	0.75	15	2:1
	18**	E	100	0.30	15	2:1
	19	E	400	1.95	15	2:1
	20	F				

1 PE=polyethylene

* Melt index of partially cross-linked polyethylenes determined after cross-linking but prior to extrusion into film.

** Comparison runs. The polyethylene in these runs had not been cross-linked.

Table IV

Elmendorf Tear Strength (g/μm)						
	Machine Direction	Transverse Direction	MD Elongation (%)	Dart-Drop F ₅₀ (g/μm)		
Run	16*	30.0	470	4.9		20
20	17	1.7	270	4.5		
	18*	5.0	190	—		
	19	3.0	55	—		
	20	1.2	54	—		

25 * Comparison runs. The polyethylene in these runs had not been cross-linked.

Example III

The procedure of Example II was repeated except that the polyethylene was extruded through an annular die having an average diameter of 25.4 mm and a die gap of 0.68 mm.

Polyethylene type G was an ethylene homopolymer having prior to cross-linking a narrow molecular weight distribution, a density of 0.955 g/cm³ and a melt index of 1.0 g/10 min.

The operating conditions of the blown film process were adjusted to form a film of about 25.4 μm in thickness. The film thickness and concentration of 2,5-dimethyl 2,5-di(t-butylperoxy)hexyne-3 were calculated as described in Example II.

Further experimental details and the results obtained are shown in Tables V and VI.

Table V

Table V						
	Poly- ethylene Type	Peroxide Concentration (ppm by weight)	Melt Index* (g/10 min)	Frost line Height (cm)	Blow- up Ratio	
Run	21**	G	none	1.0	10	4:1
40	22	G	90	0.32	10	4:1
	23	G	190	0.08	10	4:1

45 * Melt index determined on polyethylenes after cross-linking but prior to extrusion into film.

** Comparison runs. The polyethylene in these runs had not been cross-linked.

Table VI

Table VI						
	Elmendorf Tear Strength (g/μm)					
	Run	Machine Direction	Transverse Direction			
50	21*	0.92	14.0			
	22	0.88	6.0			50
	23	0.80	4.2			

50 * Comparison run. The polyethylene in this run had not been cross-linked.

Claims

1. A blown film process in which partially cross-linked polyethylene is extruded into film having a thickness in the range of 10 to 100 μm, said process having a blow-up ratio in the range of 1.2:1 to 4:1 and a MD draw down ratio in the range of 2 to 60, said polyethylene having had before cross-linking, a density in the range of 0.940 to 0.970 g/cm³ and a melt index in the range of 0.2 to 15.0

g/10 min and having been cross-linked to an extent sufficient to provide, after extrusion in said blown film process, a film having an Elmendorf tear strength in the transverse direction substantially less than the Elmendorf tear strength in the transverse direction of a film produced from the polyethylene, before cross-linking, under the same conditions as the film produced from the partially cross-linked polyethylene. 5 5

2. A process according to claim 1, wherein the film thickness is in the range of 15 to 100 μm and the MD draw down ratio is in the range of 3 to 60. 10 10

3. A process according to claim 2, wherein the film is extruded to a thickness between 25 and 35 μm and the polyethylene is cross-linked to an extent sufficient to provide a film having an Elmendorf tear strength in the transverse direction in the range of 0.4 to 10 g/ μm . 15 15

4. A process according to claim 3, wherein the polyethylene is cross-linked to an extent sufficient to provide a film having an Elmendorf tear strength in the transverse direction in the range of 0.4 to 2.5 g/ μm . 20 20

5. A process according to claim 2, wherein the film is extruded to a thickness between 35 and 100 μm and the polyethylene is cross-linked to an extent sufficient to provide a film having an Elmendorf tear strength in the transverse direction in the range of 10 to 150 g. 25 25

6. A process according to any of the preceding claims, wherein the MD draw down ratio is in the range of 6 to 40. 30 30

7. A process according to any of the preceding claims, wherein the blow up ratio is from 1.2:1 to 2.5:1. 20 20

8. A process according to any of the preceding claims, wherein the frost line height is in the range of 10 to 66 cm. 25 25

9. A process according to claim 8, wherein the frost line height is in the range of 10 to 40 cm. 30 30

10. A process according to any of the preceding claims, wherein the polyethylene is selected from the group consisting of a homopolymer of ethylene, a copolymer of ethylene and a higher α -olefin having 4 to 10 carbon atoms, and mixtures thereof. 35 35

11. A process according to claim 10, wherein the polyethylene is selected from the group consisting of a homopolymer of ethylene and a higher α -olefin having 4 to 10 carbon atoms, and mixtures thereof. 30 30

12. A blown film process according to any of runs 2, 3, 5, 6, 8, 9, 11 to 14, 17, 19, 20, 22 and 23 in the above-mentioned examples. 30 30

New Claims or Amendments to claims filed on 6 July 1979.
Superseded Claim 11.

New or Amended Claims:—

35 Claim 11 deleted; Claim 12 renumbered Claim 11. 35

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1979. Published by the Patent Office,
25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.